Electrodeposited Nano Co-P: Coating Development and Technology Insertion at NADEP-JAX

Integran Technologies Inc.

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- Rowan Technologies – Keith Legg
- Integran Technologies
  - Francisco Gonzalez, Jon McCrea, Diana Facchini, Mike Uetz
- NADEP-JAX
  - Ruben Prato, John Devereaux, John Lamkin, Ernstine Lawson

Support for the Program

- SERDP and ESTCP Program Office
- JSF Office
- Technology Partnerships Canada

Collaborators

- Goodrich, Messier-Dowty, Pratt & Whitney Canada, Smiths Aerospace

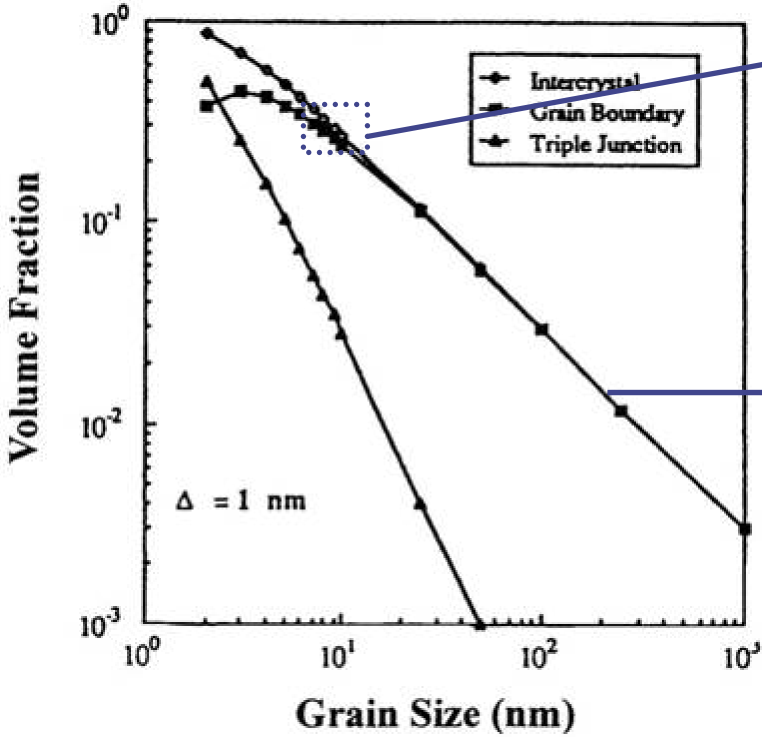
Technology Partnerships Canada

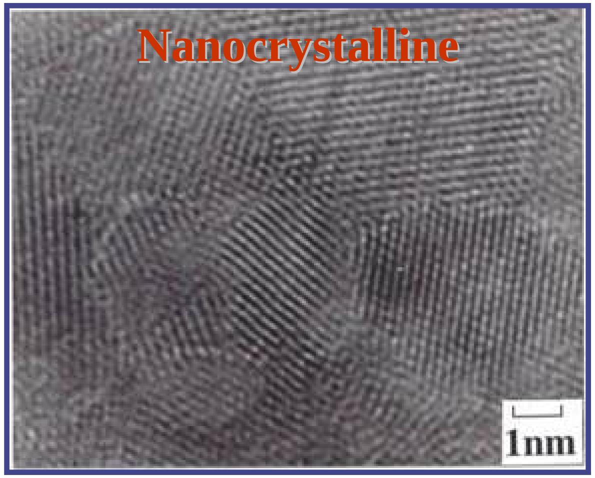
Overview

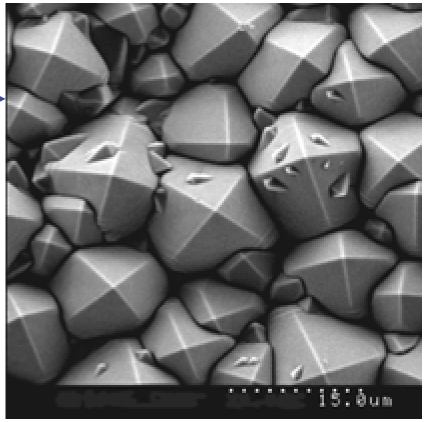
- **Nanocrystalline Materials**
- **History of the nCoP NLOS Cr-replacement Project**
- **Overview of nCoP Process and Properties**
- **Scale-up of Tank Process**
  - Installation at Integran
  - Installation at NADEP-JAX
  - Dem/Val Activities
- **Development of Selective Area (Brush) Plating**
- **Joint Test Protocol (JTP) – Sample Prep and Testing**
- **Thin Dense Chrome Development**
- **Application-specific Testing**
  - Smiths Aero
  - Messier Dowty
- **Summary**

Nanocrystalline Materials

A reduction in grain size below 100nm, significantly increases the intercrystalline content in the material, leading to many unique properties (high hardness/strength)







Need to understand the relationship between **Microstructure** and **Processing conditions**

History of Cr-Replacement Project

SERDP Project #PP-1152 – June 2000 to March 2003

Objectives: Develop an environmentally benign advanced nanocrystalline based coating technology that:

- Can be applied to non-line-of-sight surfaces
- Is compatible with conventional electroplating infrastructure
- Will produce coatings that meet or exceed the overall performance of hard chrome (hardness, wear, corrosion, fatigue)

Nano Co-P as EHC alternative was developed and demonstrated at the lab scale

ESTCP Project #PP-0411 – May 2003 to May 2006

Objectives:

- Scale up to industrial production & move to depot (Integran/NADEP-JAX)
- Develop nano Co-P selective plating as repair
- Develop nano Co-P based TDC alternative
- Define and Execute JTP

Cobalt alloy selection

- Good mechanical properties, High plating efficiency
- No constituents on EPA or AFMC lists of hazardous materials
- Longer term view

History of Cr-Replacement Project

Progression of TRL / Timeframe

Year **Project**

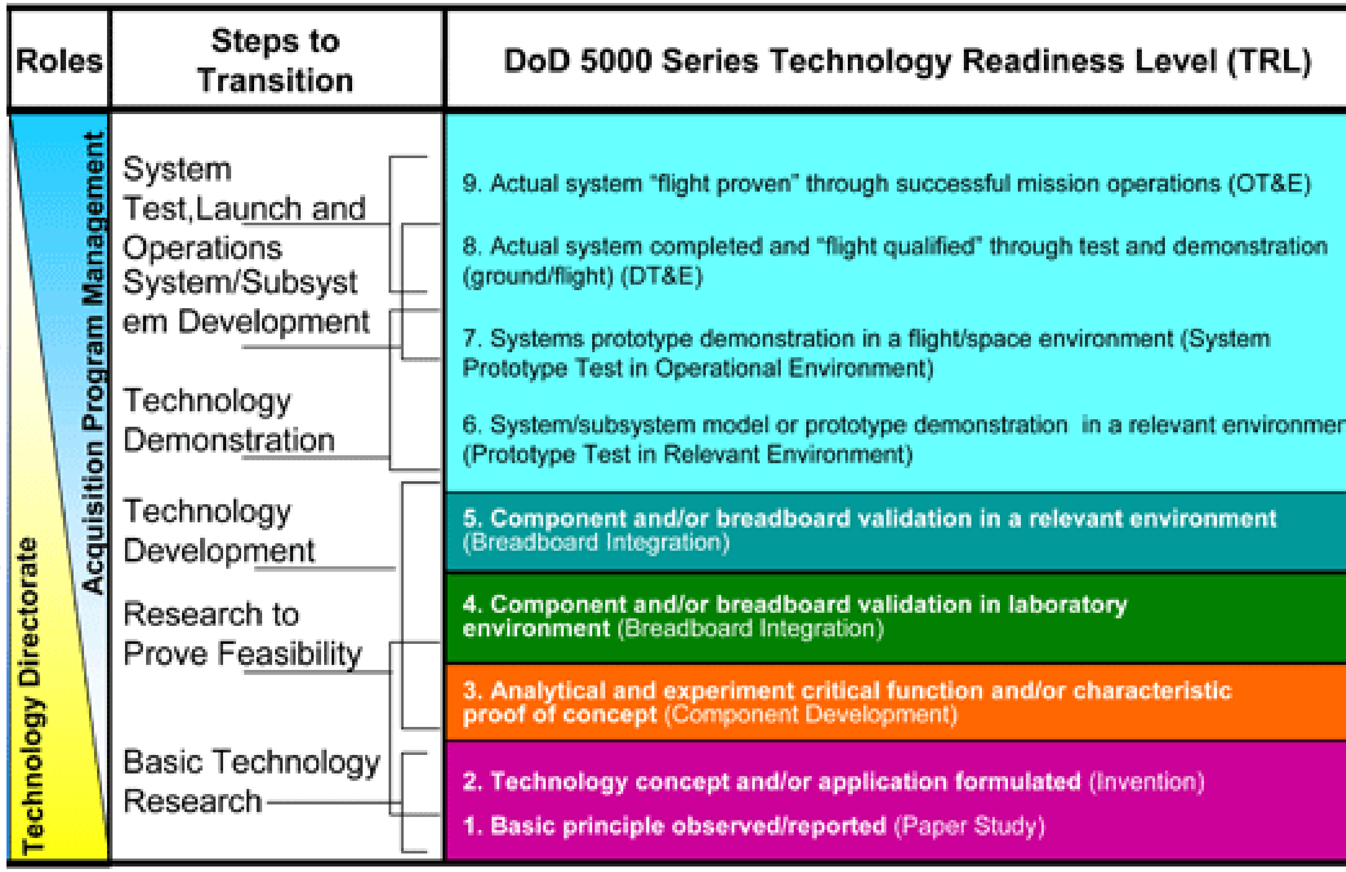
2006

ESTCP
PP-0411

2003

SERDP
PP-1152

2000



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Technology Description

Process

Simply an electrodeposition process

- Parameters modified to yield deposits with average grain sizes below 100nm using pulse plating

Plating Efficiency >90%

- High Deposition rates (2-8 mils/hr)
- 10x the plating rate of EHC at same power current density
- 1/10th the power consumption at the same plating rate

Consumable & nonconsumable anode

- Cobalt Pieces in Ti basket, Graphite

Phosphorus Content: 0 to 10wt%

- Controlled by solution chemistry and plating conditions

Technology Description

Solution Control and Maintenance

Similar to that required for nickel-plating solutions:

- **Filtration**
  - Control particles
- **pH monitoring**
  - Control deposit uniformity
- **Surface tension control**
- **Solution density monitoring**

Periodic maintenance procedures

- **Activated carbon treatment**
  - Organics
- **Dummying (low current density plating)**
  - Remove various metallic impurities

Properties Summary

		Nanocrystalline Co-P	Hard Chrome
Hardness	*As-Deposited*	600-700 VHN	800-1200 VHN
	HT @ 250°C	700-800 VHN	-
			-
Ductility		2 – 7 % Elongation	<.1%
Thermal Stability		400°C	400°C
Wear	*Adhesive (Pin-on-disk)*	5-6 x 10^{-6} mm^3/Nm (Alumina Ball on Nano Co-P Disk)	9-11 x 10^{-6} mm^3/Nm (Alumina Ball on Cr Disk)
	Coefficient of Friction	0.5	0.7
	Abrasive (Taber)	18 mg / 1000 cycles (CS-17) 11 mg / 1000 cycles (CS-10)	3.2 mg / 1000 cycles (CS-17) 1.0 mg / 1000 cycles (CS-10)
Corrosion	Salt Spray	Protection Rating 7 @ 1000 hrs	Protection Rating 2 @ 1000 hrs

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Scale up of Tank Process

Industrial Scale Up at Integran Technologies

INTEGRAN

Integran Tank

- Dimensions (3’x5’x3’)
  (~1300L)
- 1” Polypropylene
- Separate well to house pump and filter
- 3/4hp pump
- 1µm filter
- 60kw heaters (Ti) digitally controlled

Technology Partnerships Canada

Industrial Scale Up at Integran Technologies

Dem/Val Studies

- **Integran 1300 L Bath has been in operation for 26 months, >33,000 Amp-hrs, No major problems**
- **Process Verification**
  - Scaled-up process produces acceptable nanostructured coatings on small and large test coupons
  - Verified on large test pieces (4" ID, 1' Length)
- **Operating Window Identification**
  - Optimal Bath chemistry and Pulse Conditions identified
  - Temperature, Current density and pH operating ranges identified
    - Based on hardness, grain size, composition, coating uniformity
- **Contaminant Study**
  - Completed a lab scale study of the effects of various metallic contaminants in the plating solution (Cr^{6+}, Cr^{3+}, Fe^{2+}, Fe^{3+}, Cu^{2+}, Ca^{2+}, Ni^{2+}, Na^{+}, F^{-} ,and SO_4^{2-})
- **Developed activation procedures for 4340, 4130, 300M, 15-5PH, Hy-Tuf, nCoP, Al**
- **Developed alkaline dip stripping solution: avg removal rate ~0.001"/hr**

Operating Manual and Process transferred to NADEP-JAX

Technology Transfer to NADEP-JAX

Milestone: Dem/Val Tank Setup at NADEP-JAX

- Dimensions
  - 47”x30”x48”
  - approx Vol. 290 gals
- Liner Type
  - Modified Vinyl based Terpolymer
  - 0.125” thick
- Ni Plated Copper Buss Bars
- In tank pump and filter
- ¾ hp pump
- 10 micron filter
- PVDF Steam Coil, new steam lines & regulator

290 gal Dem/Val plating tank

Technology Transfer to NADEP-JAX

Milestone: Chemistry Make-up at NADEP-JAX

- Operating Vol: 250 gals
- Proprietary Bath Chemistry
- Low pH
- High Temp
- Assisted by Integran

Building of new Nano Co-P Plating Bath

Technology Transfer to NADEP-JAX

Milestone: Pulse Rectifier Installed at NADEP-JAX

- Integran's specifications
- Peak current 1500A
- Average current 500A
- Pulse timing (ton and toff)
  - 0-100ms, Δt=0.1ms
- Remote Control Micro Touch Controller
- First verified at Integran

Remote Controller

Pulse Rectifier

Technology Transfer to NADEP-JAX

Milestone: Initial Plating and Bath Optimization at NADEP-JAX

- 4130 Steel test coupons plated
- Initially dull, non-uniform deposits towards edges
    - Process parameters adjusted
- Subsequent deposits bright and uniform
- Plating rate: 5-6 mil/h

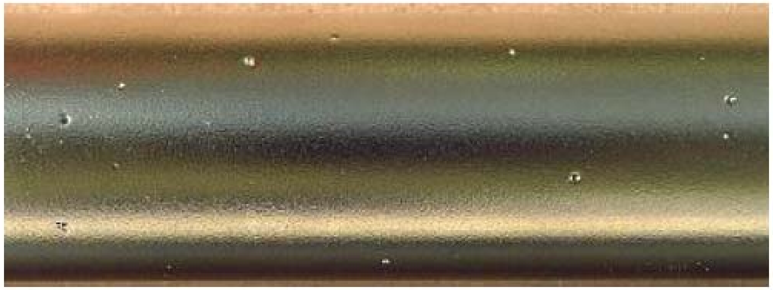
Dem/Val tank with Electrolyte and plating

First 4130 panel plated

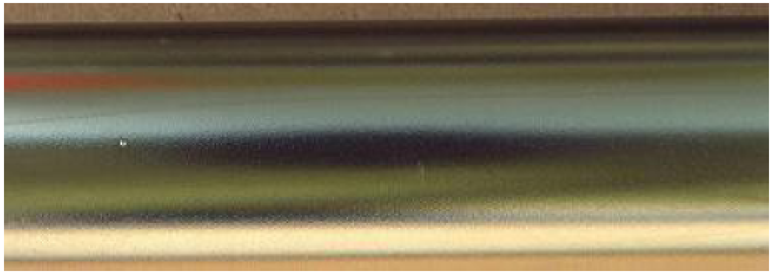
Technology Transfer to NADEP-JAX

Process Troubleshooting - Pitting

- Pitting of nano CoP observed at NADEP-JAX
  - All initially plated parts
  - Significant pitting present
- Caused by organic contamination
  - Leaching of newly installed liner from bath matrix and operating temperatures
- Carbon filtering significantly reduced pitting



Before carbon treatment



After carbon treatment

Technology Transfer to NADEP-JAX

Process Troubleshooting - Adhesion

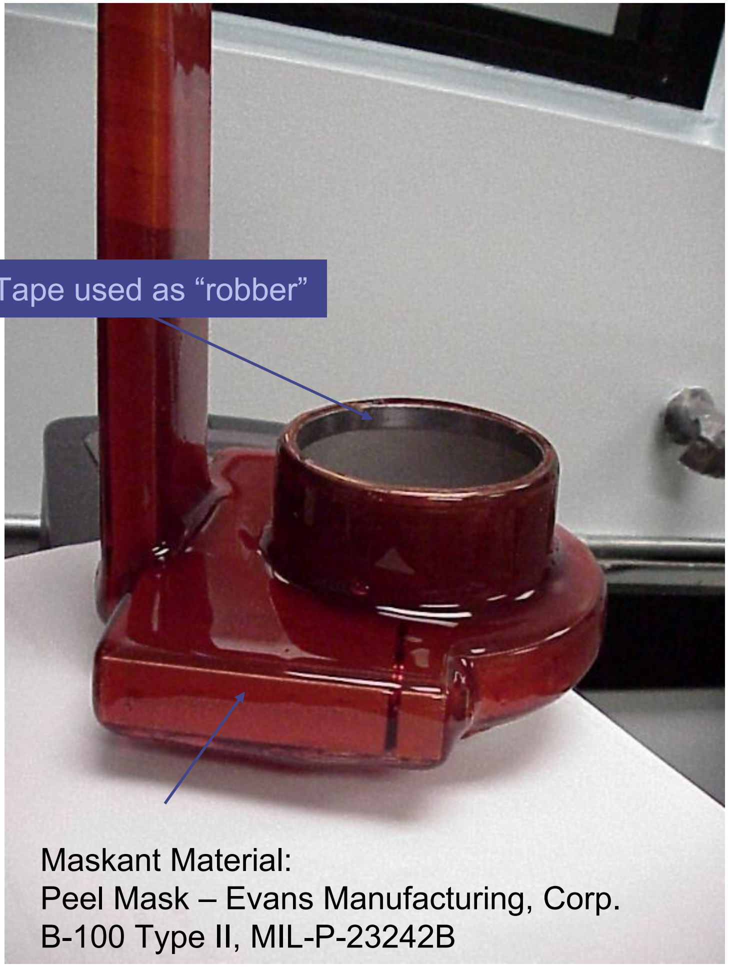
- Adhesive failures of nano CoP observed at NADEP-JAX
  - 4130 flat coupons and IDs
  - Thought to be due to activation procedure
- Alternate activation procedure developed by Integran
  - Good adhesion demonstrated on 4130 flat coupons and IDs
  - Showed promise at NADEP-JAX
- Procedure to be implemented by NADEP-JAX
  - Activation tank setup in progress
  - Retrofit of existing nearby tank
  - Requires liner, buss bar & rectifier hook up

Adhesion failures
Previously used activation

Good adhesion
Newly developed activation

Component Plating Trials at NADEP-JAX

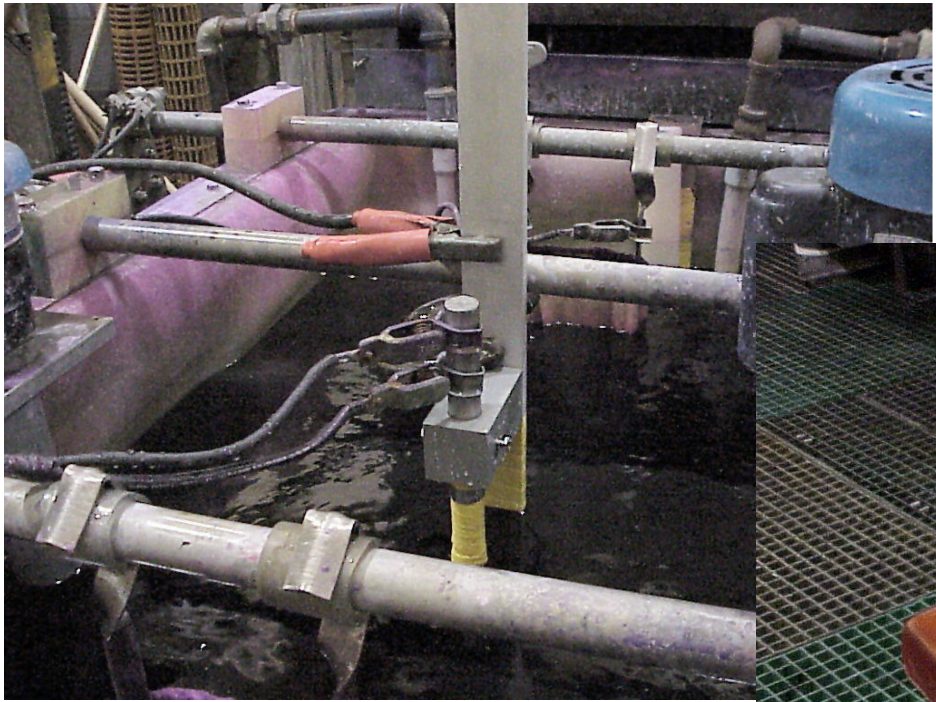
Demonstration with high temp maskant and titanium basket anode



Section from a P-3 MLG Actuating Cylinder

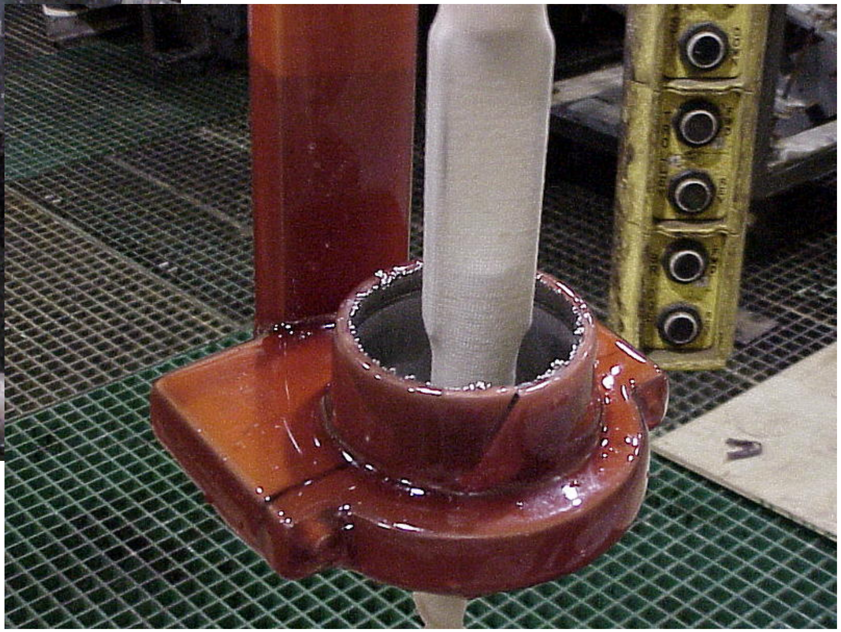
Component Plating Trials at NADEP-JAX

Demonstration on an ID section of a P-3 MLG Actuating Cylinder



nCo-P Plating on ID:
Section from P-3 MLG Actuating Cylinder

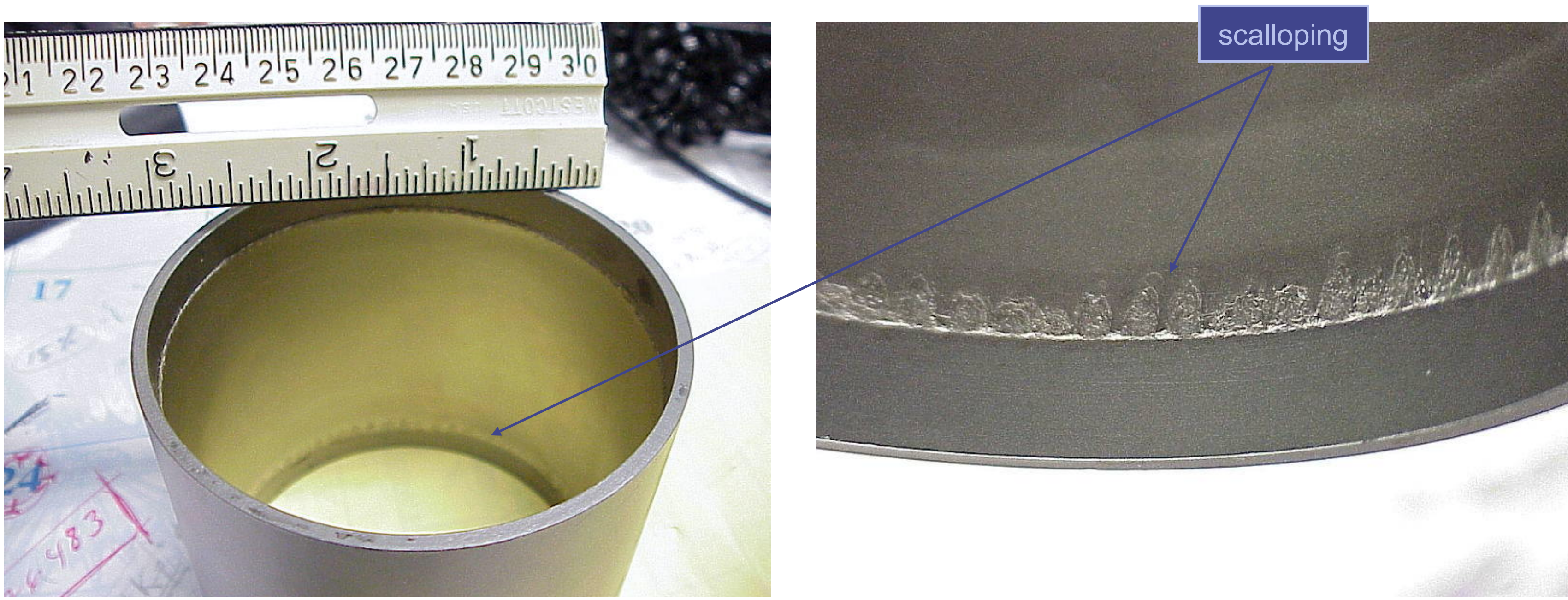
Plating Time: 4 hrs



Rack Assy after plating:
Section from P-3 MLG Actuating Cylinder

Component Plating Trials at NADEP-JAX

Demonstration on an ID section of a P-3 MLG Actuating Cylinder



Part after de-masking:
Section from P-3 MLG Actuating Cylinder

Appearance: Dull matt gray, few shallow pits
Coating Thickness: 10.0 mils /side
Deposition rate ~ 2.5 mil/hr

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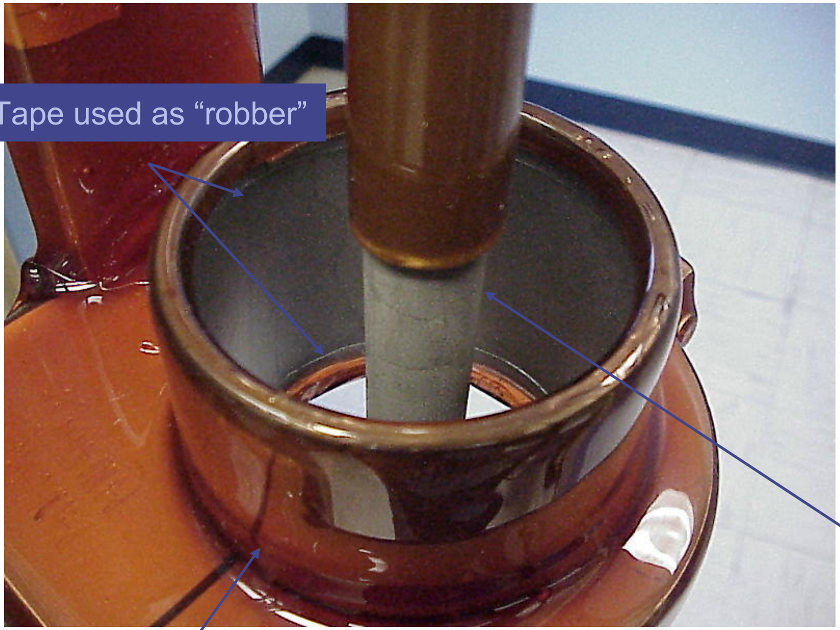
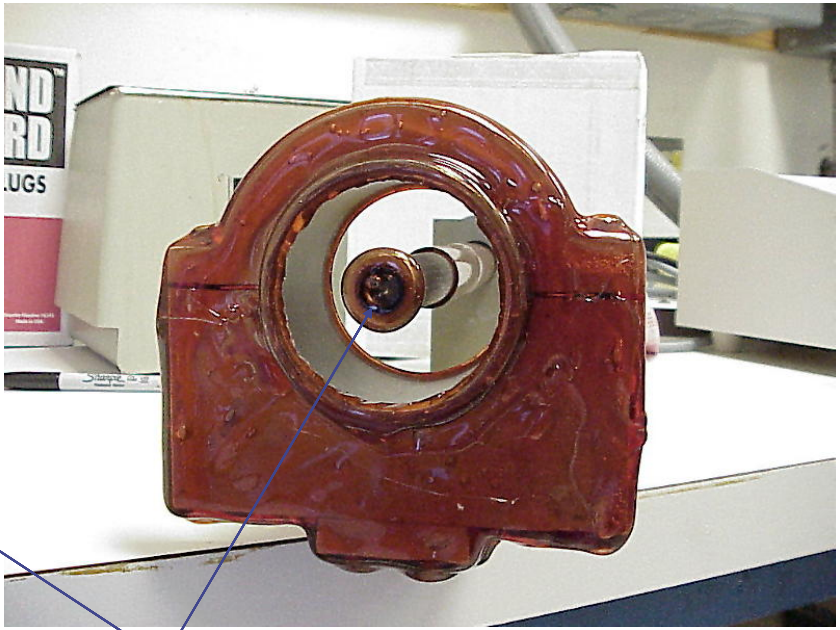
Component Plating Trials at NADEP-JAX

Demonstration with high temp maskant and Solid cobalt anode

Lead Tape used as “robber”





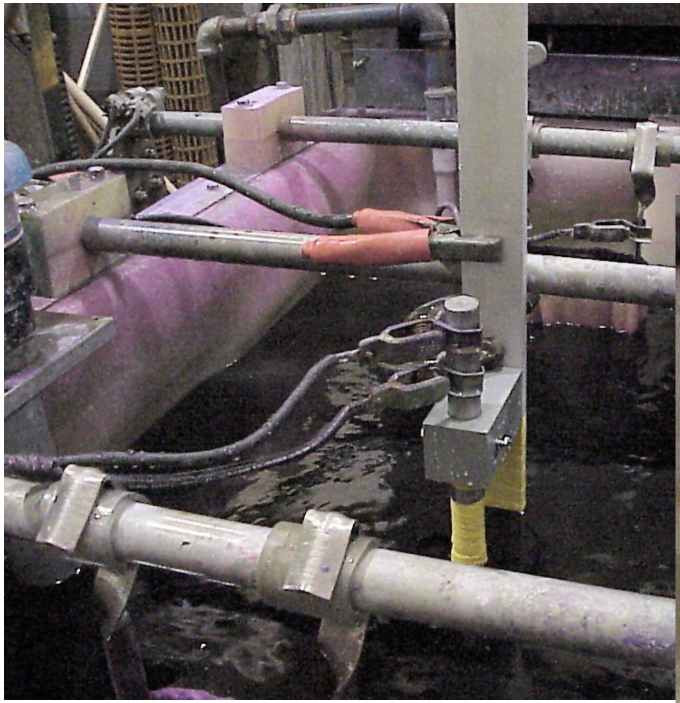
Maskant Material:
Peel Mask – Evans Manufacturing, Corp.
B-100 Type II, MIL-P-23242B

Cobalt Anode

Section from a P-3 MLG Actuating Cylinder

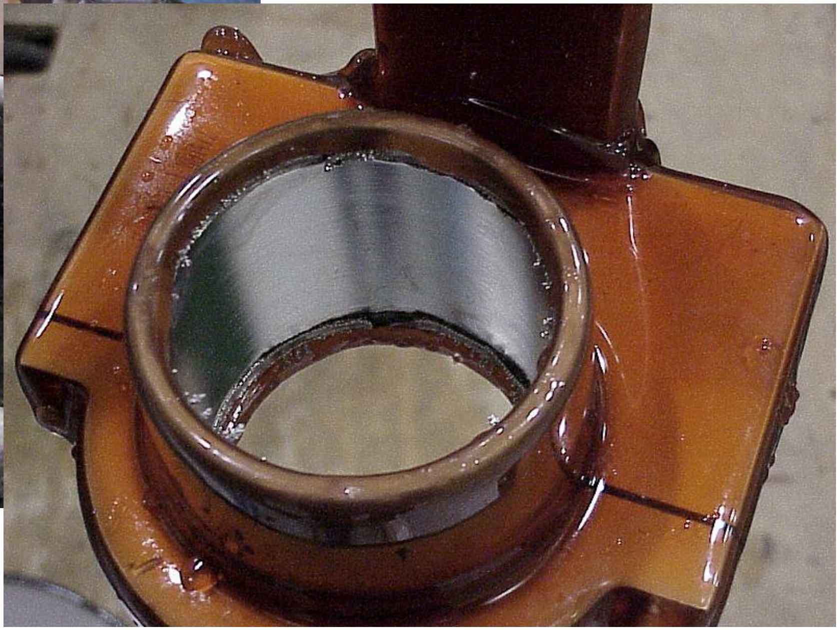
Component Plating Trials at NADEP-JAX

Demonstration on an ID section of a P-3 MLG Actuating Cylinder



nCo-P Plating on ID:
Section from P-3 MLG Actuating Cylinder

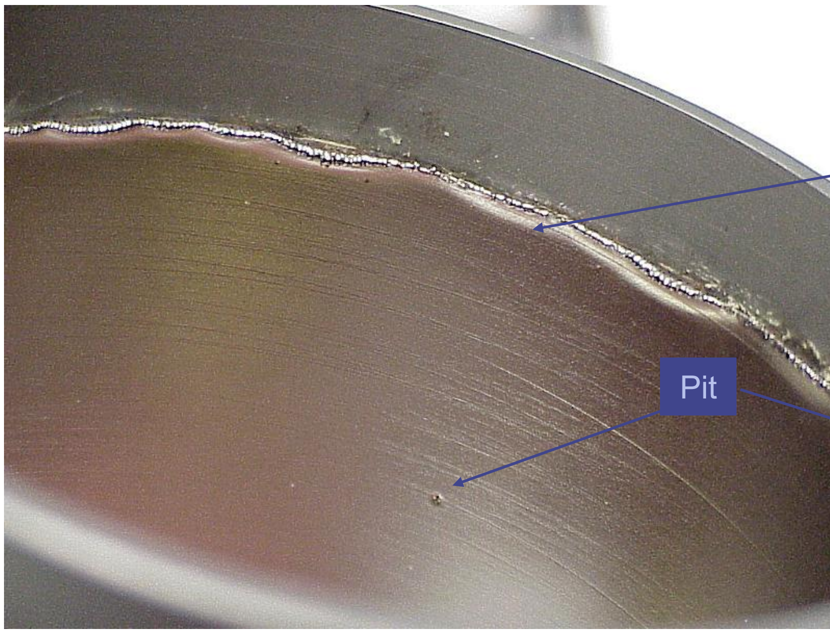
Plating Time: 2 hrs

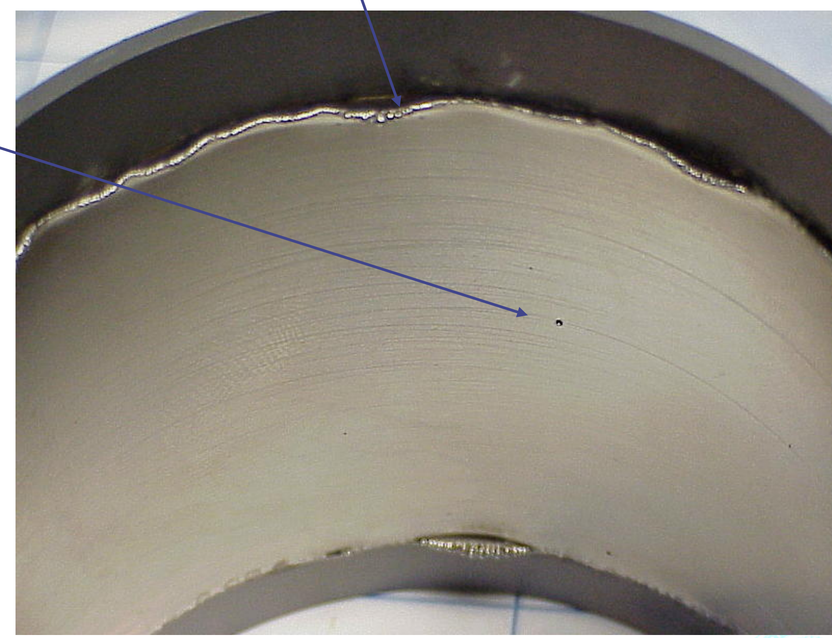


Rack Assy after plating:
Section from P-3 MLG Actuating Cylinder

Component Plating Trials at NADEP-JAX

Demonstration on an ID section of a P-3 MLG Actuating Cylinder



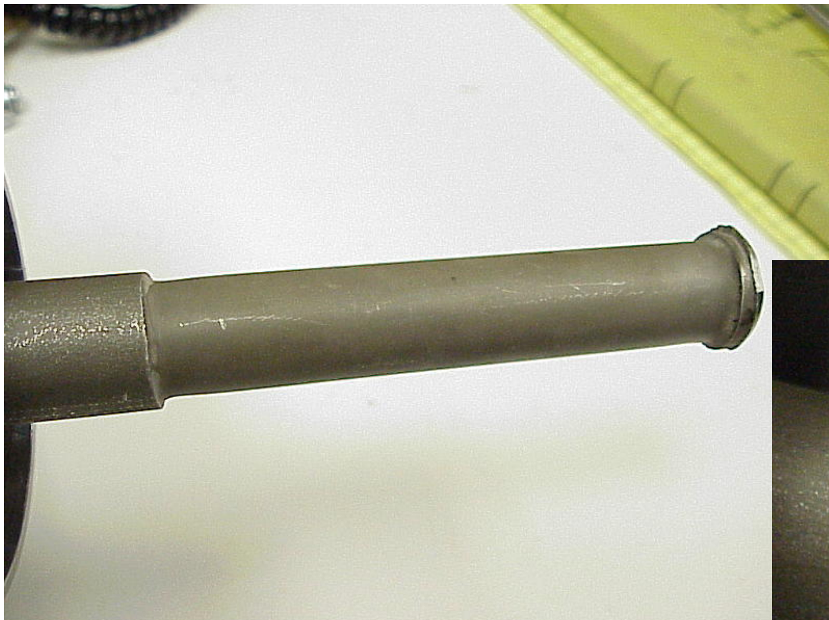


Part after de-masking:
Section from P-3 MLG Actuating Cylinder

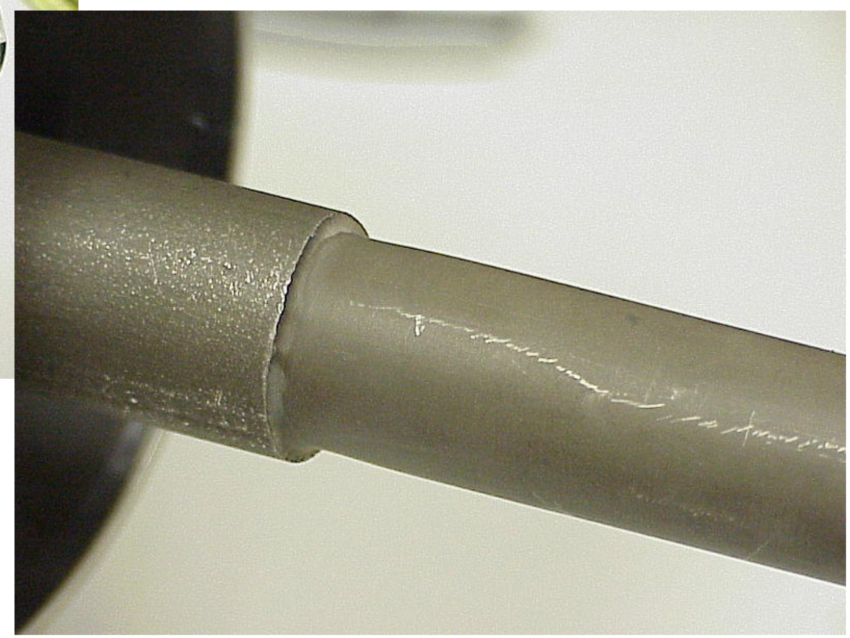
Appearance: Bright silver gray, 2 pits
Coating Thickness: 12.0 mils /side
Deposition rate ~ 6.0 mil/hr

Component Plating Trials at NADEP-JAX

Demonstration on an ID section of a P-3 MLG Actuating Cylinder



Co Plated Anode Rod after Use



Cobalt anode after 2 Hrs of plating ID Section
from P-3 MLG Actuating Cylinder

Technology Transfer to NADEP-JAX

Setup Considerations for ID Component Plating

Masking

- Crucial step due to complex geometries
  - blind areas, pin holes, dissimilar metals, etc.
- Waxes used in Cr process not applicable due to high temp of solution
- Current "Peel Mask" maskant
  - small melting pot set up in the shop
  - not as forgiving as waxes with regards to trimming
  - degradation over time causing organic contamination
- Alternative masking method prefered - Platers Tape works, but difficult with complex geometries at JAX

Anodes

- Cobalt rods
  - best performance
  - consumed very quickly
- Ti baskets with cobalt chips
  - proper anode:cathode area and adequate flow must be maintained for proper deposit characteristics

Rack Assembly of J52 Gear Spur

Technology Transfer to NADEP-JAX

Proposed Demo Parts to be Plated at NADEP-JAX

J52 Bevel Gear

P-3 MLG Actuating Cylinder

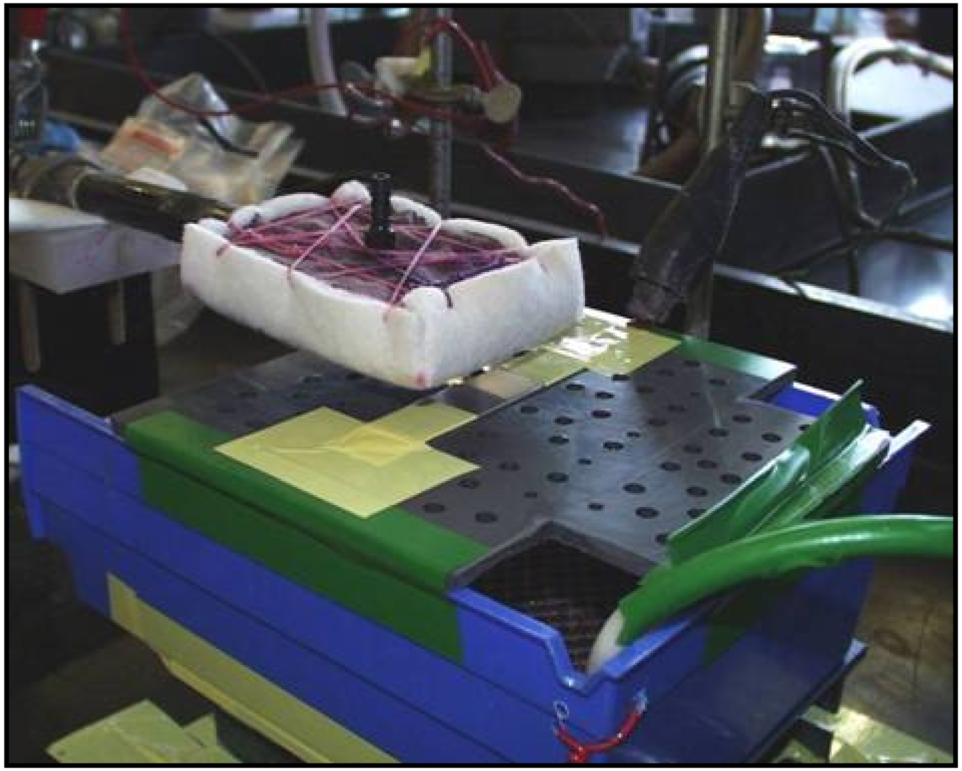
P3 MLG Cylinder Section, Axle Journal

Selective Area (Brush) Plating Development

Selective Area (Brush) Plating Development

Overview

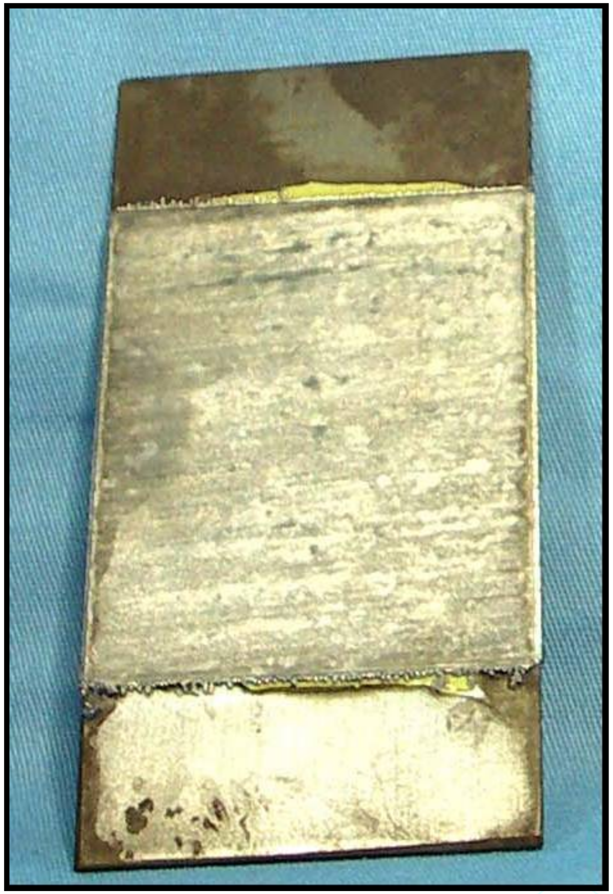
- Method to electrodeposit metal without tanks for localized coating
- Well characterized industrial process
- Moving anode – static cathode
- Can be easily field-implemented
- Can be applied manually, semiautomatic and automatic



Selective Area (Brush) Plating Development

Status

- Nano CoP brush plating procedure established
  - Optimized bath chemistry
    - Efficiency: > 90%
    - Deposition rate: 6mil/hr
    - High hardness: 600-700 VHN
    - Uniform composition
  - Good adhesion (ASTM B571)
  - Good tribological properties
  - Lower Coefficient of Friction
  - Adhesion on mild steel passes ASTM B571
- Process adapted for various geometries
  - cylindrical samples
  - large plates
  - thick deposits
- Samples being produced for JTP



Joint Test Protocol (JTP) Sample Prep and Testing

Joint Test Protocol (JTP)

JTP Summary*

- **Demonstrate adhesion on various substrates**
  - 4340, 15-5PH, Aermet 100, Al 7075 and Ti 10-2-3
- **Pre-Test Grinding Study**
- **Performance Testing (Tank plating)**
  - Corrosion (ASTM B117 & G85)
  - Rod-Seal Wear
  - Fluid Immersion
  - Hydrogen Embrittlement (ASTM F519)
  - Axial Fatigue (ASTM E466-96)

- **Performance Testing (Brush Plating)**
  - Axial Fatigue (ASTM E466-96)
  - Hydrogen Embrittlement (ASTM F519)
  - Corrosion (ASTM B117)

***Copy of JTP can be found on the HCAT website**

Pre-Test Grinding Study

Motivation

- Determine a set of reasonable (but not necessarily optimum) grinding conditions for nano CoP

Sample Preparation

- Piston Rods (1” diameter) coated with ~0.010” nano CoP at Integran
- Pre-Grinding study
  - 4 x 4130 (2 As-dep’d / 2 HT @ 191°C for 24h)
  - 2 x 15-5PH (As-dep’d)
- Final Grinding Study
  - 4340 HT to 260-280ksi
    - most susceptible or sensitive to grinding burns
    - 3 x As-dep’d
    - 3 x HT at 191°C for 24hrs
    - 3 x HT at 300°C for 1-3hrs

Four 4130 and two 15-5PH rods used for pre-grinding study

Pre-Test Grinding Study

Results

- Rods ground to ~ 0.001” at NADEP-JAX
  - Followed Mil-Std-866 using standard Al2O3 abrasives
  - Rod was easily finished to a 2-3µin roughness
  - No problems, ground very similar to chrome
- Ground coating stripped at Integran
  - Using alkaline chemical dip solution
- Stripped parts subjected to Nital Etch at NADEP-JAX
  - No evidence of any grinding burns was present

Mil-Std-866 Acceptable for nCoP

Corrosion

Sample Preparation

- Substrates
  - 4340
  - 15-5PH
  - 7075Al
- Nano CoP coating
  - 0.006”
  - 0.013”
- Post-plate grinding
  - final dimensions
    - 0.003”
    - 0.010”
  - Final surface finishes
    - 12-16 μ” Ra grind
    - <4 μ” Ra superfinish

4340

0.006” nano CoP

0.013” nano CoP

15-5PH

0.006” nano CoP

0.013” nano CoP

7075 Al

0.006” nano CoP

0.013” nano CoP

Corrosion

Testing details

- ASTM B117-9 salt fog
  - Solution: 5% NaCl
  - pH: 6.5-7.2
  - Temperature: of 35°C (95°F)
  - Test duration: 1000 h, inspect every 168 h
- ASTM G85, SO_2 salt fog
  - Solution: add SO_2
  - Test duration: 336 h, inspect at 100, 200 and 336 h
- Nano CoP to be compared to EHC of comparable thickness and surface finish (EHC samples prepared at NADEP-JAX)

Status

- Sample preparation complete
- Samples currently being ground

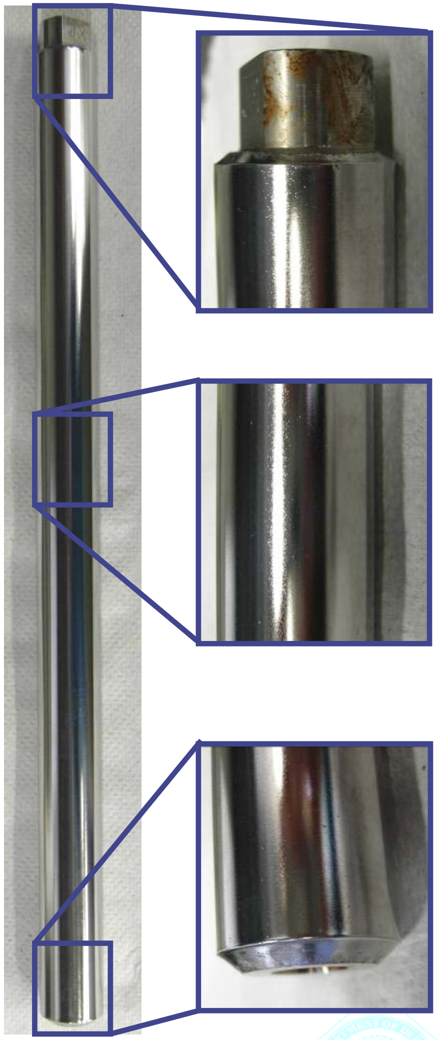
Rod-Seal Wear

Purpose

- Wear under simulated actuator service conditions
  - Compare performance of nano CoP to EHC
  - Determine optimum surface finishes on nano to minimize seal wear

Sample Preparation

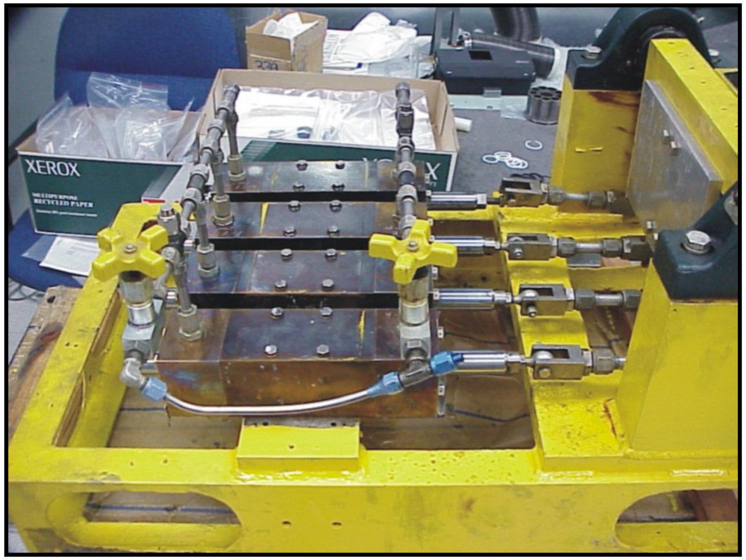
- Four hydraulic actuator rods
  - PH 13-8Mo, 16” long, 1” diameter
  - plated with 0.006-0.008” nano CoP
- Post-plating finishing
  - Rod #1: Hydrogen bake (375°F, 23h), ground to Ra 12-16 µinch
  - Rod #2: Hydrogen bake, ground to Ra 6-9 µinch
  - Rod #3: Hydrogen bake, superfinished to Ra < 4 µinch
  - Rod #4: Heat treat (300°C, 6 h), ground to Ra 6-9 µinch



Rod-Seal Wear

Testing details

- Hydraulic cylinder rod wear test
  - similar conditions to ID cylinder wear - wear against seals
  - master hydraulic piston drives four test rods
  - each rod passes through two blocks
- Two seal types to be tested per rod
  - standard MIL-P-83461 O-ring/Cap
  - Spring Energized PTFE
- Performance metrics
  - meet or exceed performance of hard chrome with a standard 12-15μ" Ra ground finish
- Testing to be conducted at NAVAIR Patuxent River



Rod-Seal Wear Test Apparatus Consisting of Four Rods Passing Through Blocks Containing Seals

Fluid Immersion

INTEGRAN

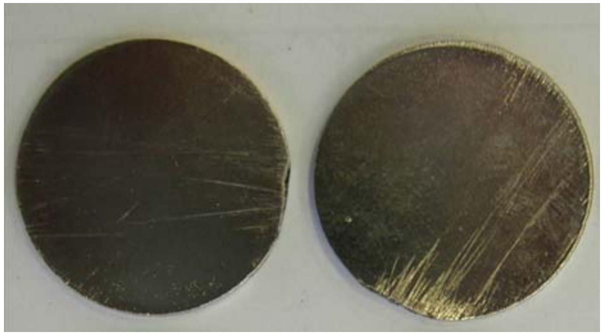
Sample Preparation

- 34 x 1" diameter 4340 discs
- Coated with 0.003" nano CoP

Testing Details

- Testing conducted at ARINC
- Coated discs submerged in various fluids
  - service conditions and overhaul fluids
- Specimens weighed before and after immersion
- Appearance of each specimen subsequent to immersion compared to that obtained prior to immersion

4340 discs
No coating



4340 discs
0.003" Nano CoP coating

Technology Partnerships Canada

ESTCP

Fluid Immersion

Preliminary Results – Visual Examination

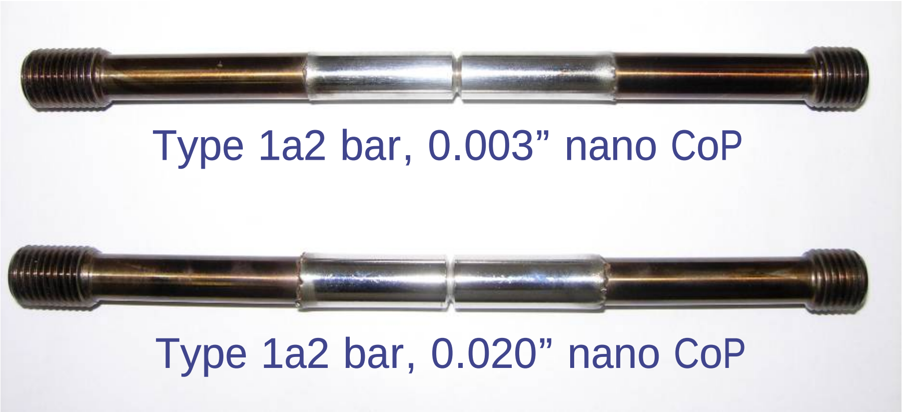
- Visual examination of specimens after immersion
- Mass changes to be examined

No effect	Discoloration
• fluorescent penetrant (for NDI) • MIL-C-87937 (aviation cleaner) • propylene glycol (de-icer) • Cee-Bee J-84A (heavy duty soak clean) • Cimstar 40 (grinding fluid)	• Nital (grind burn etch) • Oakite 90 (alkaline cleaner) • Turco Vitro-Klene (Heavy duty soak clean)
Degradation	**Not available**
• ammonium persulfate (grind burn etch) • Bleach • Turco scale Gon (descaling) • Sodium hydroxide (Cr strip) • 35% Nitric acid	• Cd plating solution (inconclusive) • Hydraulic fluids (TBD)

Hydrogen Embrittlement (ASTM F519)

Sample preparation

- Type 1a2 bar (4340, long bar)
- plated with 0.003” or 0.020” nano CoP
- Hydrogen baked at 375°F for 24h or not baked



Testing details

- Constant load testing
  - Constant load 75% of Notch Tensile Strength (NTS)

Thick.	Stress (%NTS)	Hydrogen bake	Qty const load	Results #bars >200hrs
0.003”	75%	No	4	0/4
0.020”	75%	No	4	0/4
0.003”	75%	Yes	4*	2/4
0.020”	75%	Yes	4*	0/4

Results

- Both baked and unbaked samples failed ASTM F519

Hydrogen Embrittlement - Results

ESTCP JTP and Previous Results

Test	Coating Thickness	Plating Parameters	Bar Type	Heat Treatment	Result
SERDP	0.003”	A	1a.2	None	PASS
Goodrich	0.005”	B	1a.1	None 375F/12hrs	FAIL PASS
In-House	0.005”	A	1a.2	None 375F/12hrs	FAIL PASS
Ext Customer 1	0.004”	B	1a.1	375F/24hrs	PASS
Ext Customer 2	0.005”	A	1a.1	None 375F/24hrs	PASS PASS
ESTCP	**0.003”**	**B**	**1a.2**	**None / 375F/24hrs**	**FAIL**
ESTCP	**0.020”**	**B**	**1a.2**	**None / 375F/24hrs**	**FAIL**
ESTCP(RETEST)	0.003”	A	1a.2	375F/24hrs	**PASS**

Hydrogen Embrittlement Questions

Summary

- JTP HE results first instance of failure on thin coatings (< 0.005") after baking, all previous tests passed with bake
- First tests of thick (0.020") nano CoP coatings
- Type 1a.2 bars (long) are more sensitive than 1a.1 (short)
- While change in operating parameters did not have significant effect on coating properties (structure, composition, hardness) it did have large effect on HE

Questions:

- Did change in operating parameters:
  - Somehow change deposit characteristics to let Hydrogen escape during bakeout?
  - Prevent hydrogen from getting in?
  - Does the fully dense coating prevent hydrogen from escaping?
  - How does nCoP compare to Ni / other dense non-chrome electroplated coatings?

Follow up Studies Being Conducted to Address these Questions

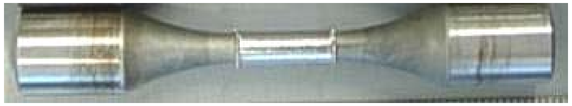
Axial Fatigue

INTEGRAN

Sample Preparation

- Peened fatigue smooth bars
  - 4340, 15-5PH or 7075 Al
- Coated with nano CoP
  - 0.006", 0.013" or 0.018" as-deposited
  - 0.003", 0.010" or 0.015" ground, Ra of 12-16 uinch

4340 fatigue bar coated with Nano CoP (as-dep'd)



15-5PH fatigue bar with Nano CoP (as-dep'd)

Testing Details

- Load-controlled constant amplitude axial fatigue testing (ASTM E466-96)
- Loads spread between high and low loads
  - High load – 85% of Fty (yield)
  - Low load – uncoated specimen fatigue life is approximately 10^6 cycles (runout)
  - maximum four points per load
- R ratio: R = -1
- Compare S-N curve to EHC coated samples of similar thickness

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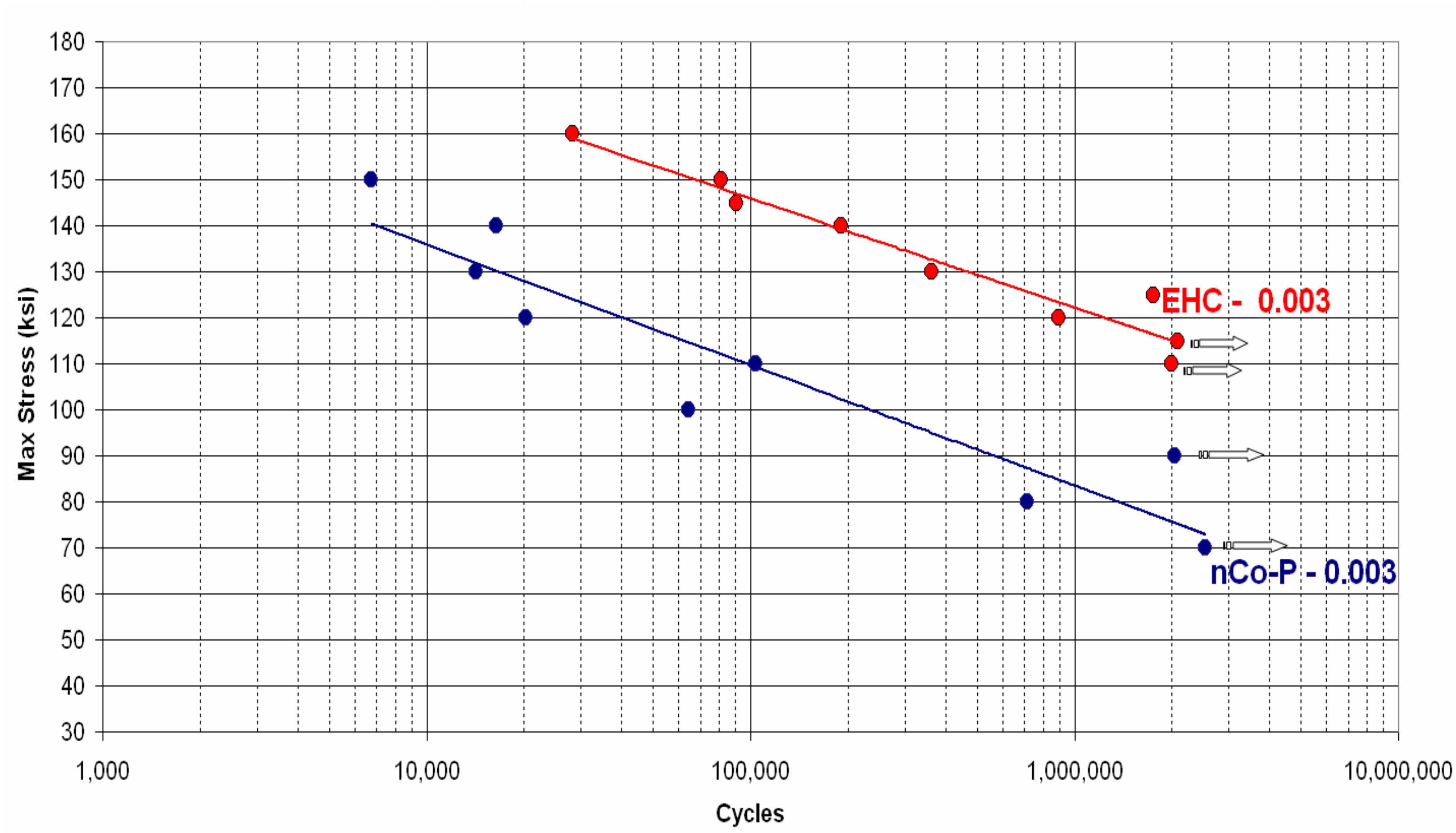
Axial Fatigue

Results – 4340 Substrate

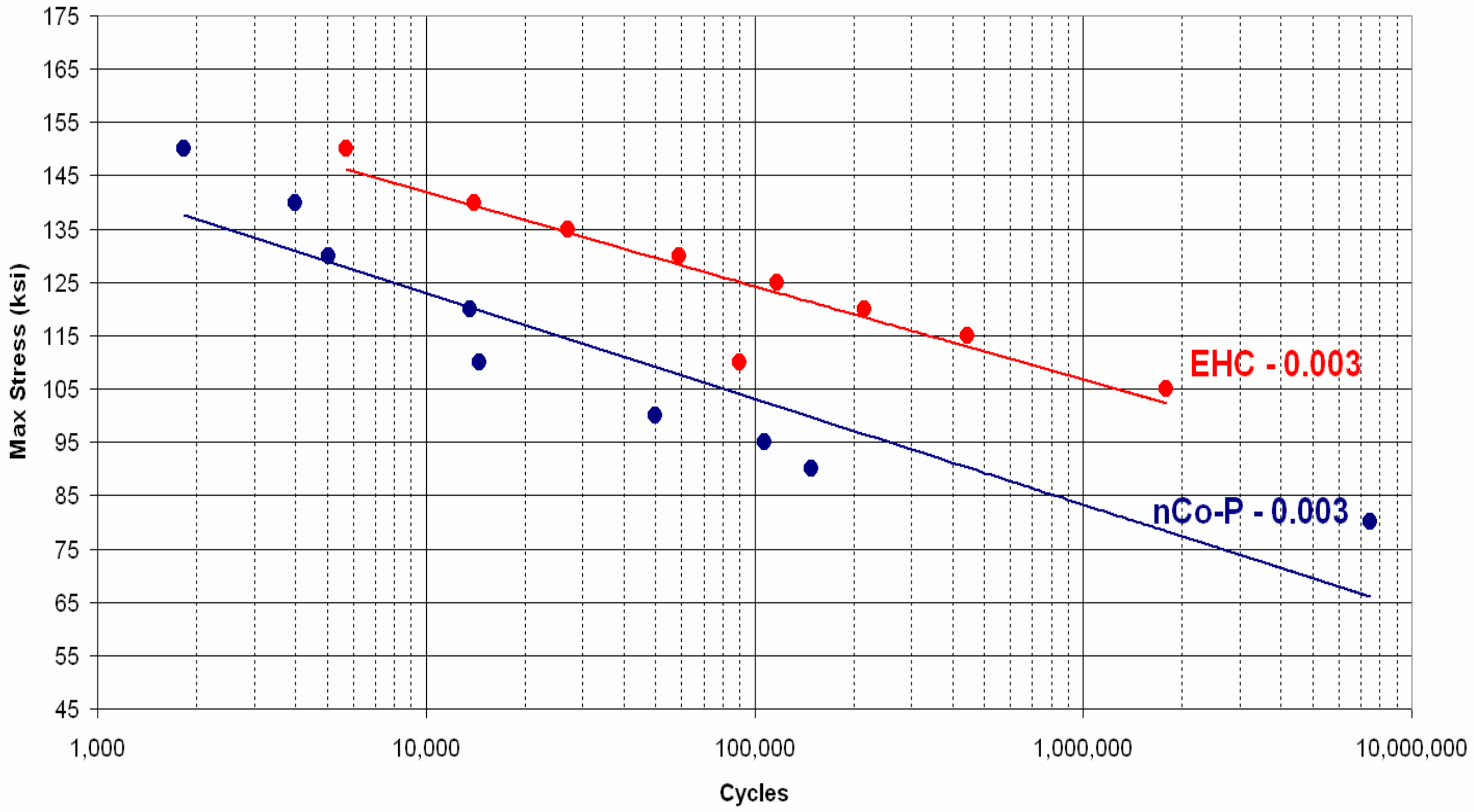
- Nano CoP shows fatigue debit compared to EHC at 0.003” and 0.010”
- Possible effects of hydrogen embrittlement?



Axial Fatigue

Results – 15-5PH Substrate

- Nano CoP shows fatigue debit compared to EHC at 0.003” and 0.010”
- Possible effects of hydrogen embrittlement?



Brush Plating Performance Testing

Testing to date - Hydrogen Embrittlement

Sample Preparation

- 8 Type 1a1 notched tensile bars (short bars)
  - brush plated with 0.003” nano CoP
  - 4 bars hydrogen baked at 375°F for 24h
  - 4 bars not baked

Testing Details

- 75% of Notch Tensile Strength (NTS)

Results

- Hydrogen baked - **PASS**
  - 1 of 4 fractured < 200h
  - Remaining bars step-loaded to >95% NTS
- Not hydrogen baked – **PASS**
  - 1 of 4 fractured < 200h
  - Remaining bars step-loaded to >95% NTS

No hydrogen bake

Hydrogen bake

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Brush Plating Performance Testing

On-going Sample Preparation

- Corrosion
  - 4”x6” peened 4340 and 15-5PH test panels
  - 0.003” and 0.010” nano CoP
- Hydrogen Embrittlement
  - Type 1a1 notched bars (short bars)
  - 0.010” nano CoP
  - With and without hydrogen bake at 375°F for 24h
- Axial Fatigue
  - Peened 4340 and 15-5PH smooth bars
  - 0.010” nano CoP (0.013” as-deposited)

Thin Dense Chrome (TDC) Development

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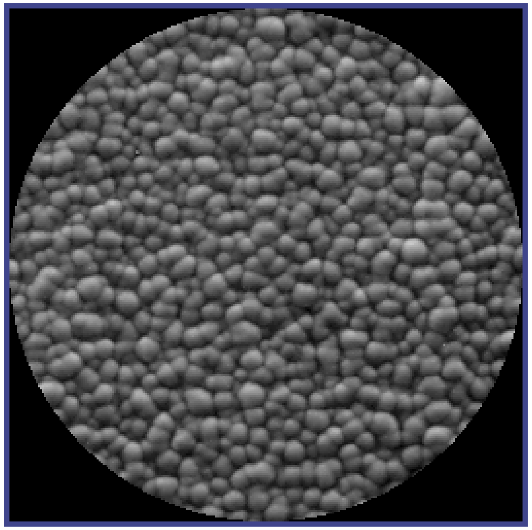
TDC Alternative Development

Investigated Range of CoP Alloys (0-12wt%P) and tested for:

Thickness Uniformity, Surface Finish, Morphology, Adhesion, Ductility, Corrosion, Hardness, Sliding and Abrasive Wear Testing

(Benchmark comparison made against TDC (AMS 2438A)

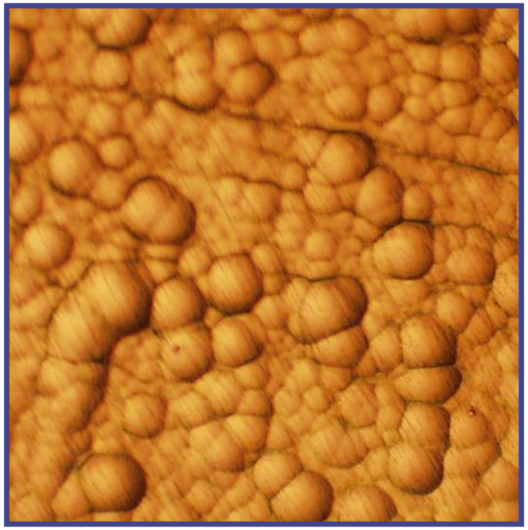
Surface morphology is **nodular** (similar to that of Thin Dense Chrome)



Thin Dense Chrome

Hardness

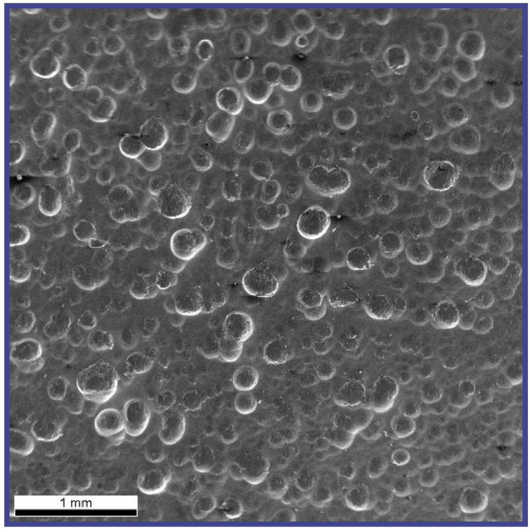
>900VHN



Co 2-3wt% P

Hardness

~600-650VHN As-Dep



Co 11wt% P

Hardness

~700VHN As-Dep

TDC Alternative Summary

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Two Classes of Coatings Recommended

	Class 1 nCoP 2-3wt%P HT 191°C – 24hrs	**Class 2** nCoP 11-12wt%P HT 300°C – 6hrs
Application Types	When corrosion resistance is required and the substrates cannot be HT to 250°C	When corrosion resistance is not required and the substrates can be HT above 250°C
Thickness Uniformity	Need proper masking/shielding to achieve required thickness	
Surface Finish	Surface roughness unaltered after coating to 0.0005”	
Adhesion	Pass	Pass
Ductility	4-5%	~1%
Corrosion Salt Spray	Pass	Fail
Hardness	650 VHN	1100 VHN
Wear (Sliding)	Good	Good
Wear (Abrasive)	Good ~18mg/1000cycles	Very Good ~7.7mg/1000cycles

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Application-Specific Testing

TDC Alternative Dem/Val

Motivation

- Part of Dem/Val to qualify nano CoP as TDC alternative

Objectives

- Demonstrate nano CoP for use as TDC alternative in actuator cylinder

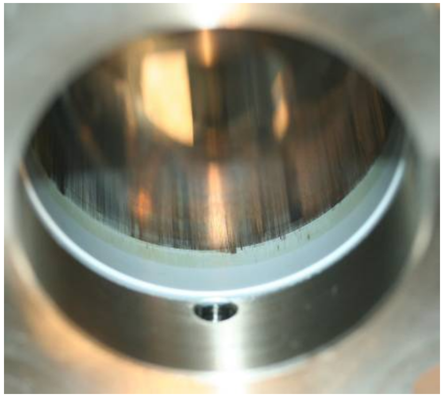
Methods

- Actuator cylinder IDs coated with 0.0003” nano CoP
- Endurance testing and evaluation at Smiths Aerospace
  - Compared to Armoloy NTDC in a side by side rig test

TDC Alternative Dem/Val

Preliminary results

- **Endurance testing and evaluation**
  - Nano Co-P showed similar behaviour and performance to Armoloy NTDC
  - Considered equivalent in this application
- **Future Work**
  - More extensive testing of nano CoP required for this application
  - Commercial details
    - Local suppliers of this process?
    - Would like more information of production quantity processing costs from a more dedicated, production facility



TDC-coated actuator
Post-test

nano CoP-coated actuator
Post-test

Endurance Testing

Motivation

- TDC alternative for NLOS applications in landing gear manufacturing

Objectives

- Demonstrate adhesion on base metal substrates
- Design and fabricate plating anodes for plating 1" ID cylinders
- Assess performance of nano CoP coatings in utility actuator application

Methods

- Nano CoP (0.0005") applied to
  - OD surface of actuator piston rods
  - ID surface of actuator cylinders
- Endurance testing at Messier Dowty
  - 20,000 cycles
  - simulates a scheduled commercial overhaul inspection

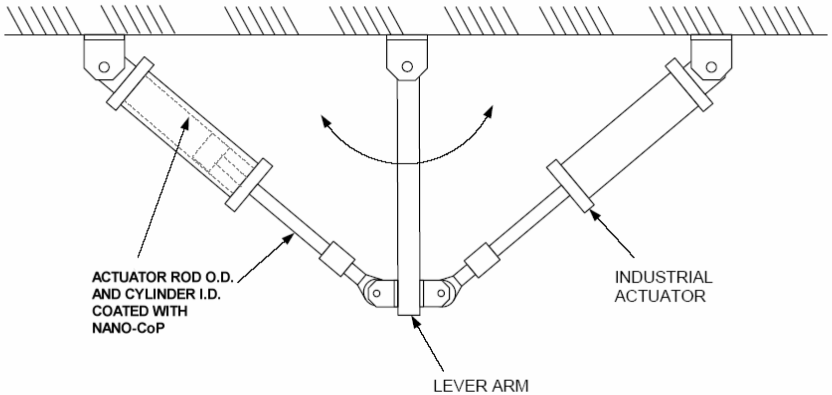


FIGURE 1 – ILLUSTRATION OF TEST SETUP

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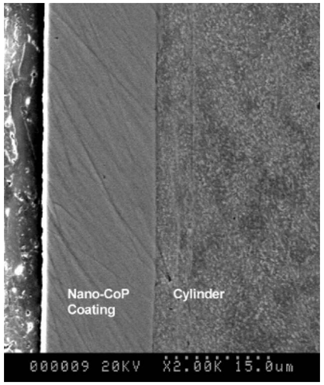
Endurance Testing

Performance Testing and Results

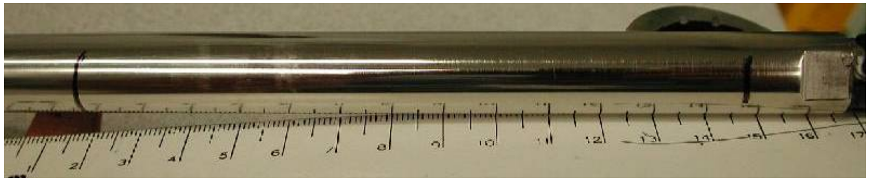
nano CoP coating

- conformed to substrate surface
- no change in surface roughness (Ra = 0.06)



- 1 of 3 cylinders showed delamination during sectioning
  - caused by activation procedure
  - corrective actions implemented

Endurance testing



- Minor wear/scoring (discoloration)
  - localized areas of piston and cylinder
  - sub-micron depth scoring (negligible wear)

- Acceptable leakage performance
  - <1 drop of hydraulic fluid in 25 cycles

- tests successful in meeting performance goals
- coating under consideration for the A 380 nose landing gear design team

Summary

- **Industrial scale-up of nCoP process successful to date**
- **NADEP-JAX Dem/Val**
  - Tank installation complete
  - Technology transfer underway
  - Some growing pains incurred (i.e. issues with Pitting/Adhesion/Anode Set-up)
  - Issues have been addressed Dem/Val process continues
- **JTP Testing**
  - Sample preparation and testing well underway
  - Initial hydrogen embrittlement issues seem to be resolved
  - Additional studies being performed to get to root of HE Problem
  - Fatigue testing to be repeated
- **TDC Alternative Development**
  - Preliminary testing compete
  - Application-specific testing ongoing
- **Application-specific testing**
  - Actuator rig testing reveals good results against polymeric/rubber seals

The End

THANK YOU FOR LISTENING!

History of Cobalt Use

Cobalt is widely used in industry for years:

- **Pigments**
  - Cobalt coloring of ceramics has been known for well over 2,000 years in Persia and Egypt
- **Metallurgical**
  - Superalloys, Hastalloy, Stellite, Thermal Spray Alloys, Co-WC
- **Electronics**
  - Samarium cobalt hard magnets
  - Magnetic particle recording
  - Perpendicular recording with Co-Cr
- **Catalysts**
- **Medicine**
  - Co-Cr alloys in prosthetics
  - Cobalt 60 used in food and medical sterilisation
- **Agriculture**
  - Animal feed to increase vitamin B12 in meat
- **Plating**

Cobalt Health Concerns?

Cobalt in small amounts is essential to many living organisms, including humans

- Cobalt is a central component of the vitamin cobalamin, or vitamin B-12.
- Having 0.13 to 0.30 mg/kg of cobalt in soils markedly improves the health of grazing animals.

Health Studies

- Co(II) added to a brand of beer in the 1960, increased rate of cardiomyopathy (later attributed to poor diet combined with Co ingestion
- Diamond polishers exposed to Co, WC, etc metal dust developed chromic fibrotic lung disease, however workers exposed to cobalt in strictly Co-refining showed no evidence of lung fibrosis
- No human case studies have indicated Co alone to be potential carcinogen

American Cancer Society Has Four Categories of Carcinogens:

1. "Known to Be Human Carcinogens“

- Cadmium and cadmium compounds
- Chromium [VI] compounds
- Nickel compounds

2. "Probably Carcinogenic to Humans"

3. “Reasonably Anticipated to Be Human Carcinogens"

- Cobalt Sulfate

No chemicals used in Nano CoP Process on the ACS list of Carcinogens